United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program

SOP No.: PDP-QC-14		Page 1 of 11
Title: Quality Assurance/Quality Control (QA/QC) for Finished Drinking Water		
Revision: 2	Replaces: 10/01/01	Effective: 01/01/03

1. Purpose:

To provide standard procedures for quality assurance (QA)/quality control (QC) of finished drinking water method validation and the receipt, storage/preparation, analysis, reporting, and disposal of USDA, AMS Pesticide Data Program (PDP) finished drinking water samples.

2. Scope:

This standard operating procedure (SOP) shall be followed by all analytical laboratories conducting pesticide residue studies for finished drinking water. Sampling shall be performed by designated water treatment facility personnel under established sampling protocols (refer to SOP PDP-SAMP-PROC-08). All samples shall be split into three one-liter amber glass bottles and shipped via overnight courier to the analytical laboratory. All samples shall be filtered within 48 hours of collection if filtration is employed, extracted within 96 hours of collection, analyzed for the required compounds identified in the applicable SOP PDP-QC-13AB, and reported electronically to USDA AMS.

3. Outline of Procedures:

5.1 Method Validation
5.2 Sample Receipt
5.3 Sample Storage/Preparation
5.4 Sample Analysis
5.5 Data Reporting
5.6 Sample Disposal

4. References:

- SOP PDP-SAMP-PROC-08, "Collection, Packaging, and Shipping of Finished Drinking Water Samples, revision 1, October 1, 2001
- USDA/New York Department of Agriculture & Markets/New York City Water Authority Meeting, April 25, 2001

- ? USDA/California Department of Food and Agriculture Laboratory Meeting, February 22, 2001
- ? USDA/New York Department of Agriculture and Markets Laboratory Meeting, February 7, 2001
- ? AMS/USGS Meeting, Los Angeles, California, November 13, 2000
- ? AMS/USGS Meeting, Albany, New York, November 6, 2000
- ? USDA/AMS PDP Federal/State Meeting, October 31-November 2, 2000
- ? EPA Unregulated Contaminants Monitoring Rule (UCMR) Assessment Sampling Instructions, September 17, 1999
- ? Field Guide for Collecting and Processing Stream-Water Samples for the National Water Quality Assessment Program (Larry Shelton, USGS; Report 94-455; 1994)PDP Program Plan January – June 2001
- ? Memo from Cathy Eiden, EPA/OPP/HED, to Tom Gilding, ACPA, Follow-up to October 28, 1998, Work Group Meeting
- ? PDP/Drinking Water Monitoring Work Group Meeting, October 28, 1998
- ? PDP/Drinking Water Monitoring Work Group Meeting, September 8, 1998

5. Specific Procedures:

This standard operating procedure (SOP) represents minimum PDP requirements for QA/ QC of finished drinking water method validation and the receipt, storage/preparation, analysis, reporting, and disposal of USDA AMS finished drinking water samples. Each analytical laboratory shall have written procedures that provide specific details concerning how the procedure has been implemented in the laboratory.

5.1 Method Validation

a. Verification of Limits of Detection (LODs) and Limits of Quantitation (LOQs)

1. Follow Verification of LOD Section 6.5 of SOP PDP-QC-10, Determination of LOD and LOQ for Chromatographic Methods.

2. Prepare summary form(s) of the acquired data, including the verified LOD and LOQ values.

b. Determination of Consistent Instrumental Response

1. Standards containing the analytes specified in the applicable SOP PDP-QC-13AB shall provide consistent responses at levels equivalent to LOQ, 5xLOQ and 10xLOQ on the primary injector and detector system. If additional injector and detector combinations are to be used for quantitation, they must be likewise evaluated. For each analyte specified in the applicable SOP PDP-QC-13AB, run, at a minimum, a three point standard curve from LOQ to 10xLOQ. Run this curve three times over three days for a minimum of nine points.

2. Calculate the mean response for each level. Calculate the correlation coefficient, R, based upon these mean values.

3. Prepare summary form(s) of the acquired data.

c. Determination of Method Performance

1. Fortified samples are to be run through the entire analytical method on the primary injector and detector system. If additional injector and detector combinations are to be used for quantitation, they must be likewise evaluated.

2. Fortify samples in triplicate at approximately LOQ, 5xLOQ, and 10xLOQ for each analyte specified in the applicable SOP PDP-QC-13AB. Process these fortified samples through the entire analytical method (treat as a set of nine samples). A matrix blank (tap water) shall be subjected to the analytical method along with the fortified analytes.

3. For each data point, calculate the Percent Recovery compared to known standards to three significant figures if greater than 100% or to two significant figures if less than 100%.

4. Calculate the mean percent recovery for each level. Calculate the Coefficient of Variation (%CV) for each level, using the nine recovery values generated from LOQ to 10xLOQ.

5. Prepare summary form(s) of the acquired data by analyte and level. Refer to SOP PDP-QC-04 for PDP acceptance criteria of this acquired data.

d. Precision and Accuracy Data Collection

1. Precision and accuracy data collection shall be compiled for each analyte specified in the applicable SOP PDP-QC-13AB. Each compound shall be spiked at 2xLOQ and evaluated using a minimum of seven data points.

2. The required data points shall be obtained from:

a. 2xLOQ data points completed after Determination of Method Performance

and/or

b. Data points from laboratory spikes analyzed concurrently with samples.

3. For each data point, calculate the Percent Recovery compared to known standards to three significant figures if greater than 100% or to two significant figures if less than 100%.

4. Calculate the Coefficient of Variation (%CV) for each analyte.

5. Prepare summary form(s) of the acquired data. Refer to SOP PDP-QC-04 for PDP acceptance criteria.

e. Method Validation Reporting

1. The methodology, method validation records, summary form(s), and any other raw data generated during method validation shall be maintained by the Quality Assurance Unit (QAU).

2. Completed summary form(s) shall be submitted to the PDP Technical Director.

5.2 Sample Receipt

a. Monthly sampling plans designating sampling dates and sites shall be provided to the analytical laboratory.

b. All sample collection bottles, which contain the appropriate dechlorinating agents, will be delivered ready for use to the designated collection site.

c. Samples shall be shipped by overnight courier to the laboratory.

e. Those samples received in a damaged (e.g., warm to the touch or leaking) condition shall be discarded and not analyzed. Condition and disposal shall be recorded on the Sample Tracking Form (which accompanies the sample) and any other applicable documentation. If a sample must be discarded, the laboratory shall immediate notify the USDA AMS Monitoring Programs Office and the USDA sampling collector contractor.

f. The laboratory shall maintain a log of samples received. Refer to SOP PDP-LABOP-01, subsection 5.1.

5.3 Sample Storage/Preparation

a. If filtration is employed, each sample, shall be filtered within 48 hours of collection. Filtration may be omitted if the laboratory provides data showing this to be unnecessary for obtaining accurate results. The data shall consist of:

1. Verification of the limit of detection – spiking each compound in duplicate and carrying through entire analytical procedure.

2. Collection of seven data points at two times the limit of quantitation for each compound – may be performed concurrently with sample analysis.

c. Samples shall be extracted within 96 hours of collection. Samples shall be refrigerated until time of extraction and sample extracts shall be refrigerated or frozen until time of analysis.

d. Samples shall be extracted following the laboratory's internal procedures for all appropriate analyses.

5.4 Sample Analysis

a. Sample Set Requirements

1. Each set shall consist of those analytical samples and associated field and laboratory QA/QC samples that can be reasonably extracted in a single day and that can be run on the appropriate instruments without degradation of instrument response.

2. All components of sample sets shall be subject to the entire analytical test process as detailed in approved methodology.

3. Laboratory QA/QC samples are required for each analytical set and include a matrix blank (tap water) and appropriate matrix spikes.

4. The matrix spike(s) shall be prepared at approximately 2xLOQ with the identified marker pesticides. Refer to the applicable SOP PDP-QC-13AB. Marker pesticides will be specified in conjunction with the PDP Technical Director following completion of method validation requirements. Marker pesticides shall be chosen in order to adequately characterize the extraction and analytical processes employed by each laboratory.

5. Each analytical sample shall be spiked with the appropriate process control(s) at approximately 5xLOQ.

b. Analytical Requirements

1. Each sample shall be analyzed for the compounds specified in the applicable SOP PDP-QC-13AB.

5.5 Data Reporting

a. All concentration values shall be reported to at least two significant figures in terms of parts per billion (ppb) or parts per trillion (ppt).

b. All data shall be transmitted electronically according to established procedures.

5.6 Sample Disposal

a. Any reserve samples shall be disposed of after 96 hours of receipt, as sample analysis must occur within this timeframe.

b. Any reserve extracts, where available, may be disposed of when all requirements for acceptability criteria have been met and results have been successfully transmitted to USDA, AMS, Monitoring Programs Office.

c. Disposal shall be documented [e.g., freezer log, sample log, extraction worksheet, Remote Data Entry (RDE) chain of custody inputs] and shall

contain a minimum of date of disposal, sample number, and initials of the individual who discarded the sample.

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Revision 2	October 2002	Monitoring Programs Office

? Removed field blank requirements from sample receipt and storage/preparation procedures (subsections 5.2 and 5.3)
? Modified sample preparation procedures to allow for differential testing of each sample bottle according to internal laboratory procedures (subsection 5.3)